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MANUFACTURE AND PROPERTIES OF A CELLULOSE PRODUCT (MAIZOLITH) FROM CORNSTALKS AND CORNCOBS

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MANUFACTURE AND PROPERTIES OF A CELLULOSE PRODUCT (MAIZOLITH) FROM CORNSTALKS AND CORNCOBS

ABSTRACT

Maizolith is a dense, hard, bonelike substance made by cooking cornstalks or corncobs with caustic soda, washing the residue, beating it to a jell, and drying. The finished product has a modulus of rupture of about 16,000 lbs./in.^{2} and a volume resistivity of 3×10^9 ohms/cm^3. It is practically unaffected by oil, but becomes soft on prolonged soaking in water.

The optimum conditions for the manufacture of maizolith were found to be those indicated by the following process: Digest the shredded cornstalks three hours at 40 pounds pressure with 10 per cent of their bone-dry weight of caustic soda; dilute to a 1 per cent solution; wash free of cook liquors; dilute the pulp to a consistency of 4 per cent, beat for two hours in a combined beating engine and Jordan refiner and dry at a temperature of 70° C.

Cost figures for the process indicate that in a plant large enough to produce 5 tons per day, finished maizolith can be made for about $240 per ton, using cornstalks as the base material.

Maizolith could probably be made at a lower figure than $240 per ton as a by-product of the insulating wall board industry, by utilizing fibers or pith particles which are too fine or too highly hydrated to be used in insulating board.

By utilizing the soft residue from the process of extracting adhesive material from corncobs maizolith could probably be produced at a lower figure than that from either of the processes outlined above.

CONTENTS

I. INTRODUCTION

The work on maizolith is a part of the program on the utilization of agricultural wastes which has been carried on for the past 10 years in the chemical engineering department at Iowa State College under the direction of Dr. O. R. Sweeney. Since August, 1927, the National Bureau of Standards has been cooperating with the Iowa State College in the development, on a semicommercial scale, of two or three of the more promising processes for the utilization of farm wastes.

The purpose of the work recorded in this paper was to determine the optimum conditions for the production of maizolith, to make a study of its properties, and to get an idea of the cost of producing the material.

Maizolith is a dense, hard, bonelike material, ranging in color from a golden tan to a deep ebony. It is somewhat heavier and stronger than the hardwoods, and is a good electrical insulator. Figure 1 gives an idea of the way in which the material may be machined and polished.

Maizolith closely resembles cellulith, a material which is made from sulphite pulp, and which was patented in 1899 by Heinrich Brunswig.[1] Its properties also coincide closely with those of vulcanized fiber, a material which is used extensively in this country to-day. It is not surprising that the properties of maizolith closely resemble those of vulcanized fiber since the two products are chemically similar; that is, products which are obtained by drying gelatinized cellulose. In the case of vulcanized fiber the cellulose is gelatinized by chemical means, in the case of maizolith, by mechanical means. Any differences found in the properties of the two substances are no doubt due to variations in the raw materials and in the way the gelatinized cellulose is handled in converting it into the finished products. The following brief descriptions of the manufacture of vulcanized fiber and of maizolith will illustrate how the gelatinized cellulose is handled in the two processes.

II. METHOD OF MANUFACTURE

In the preparation of maizolith, cornstalks which have been shredded to lengths of 1.5 to 3 inches (or corncobs roughly broken up) are digested about three hours under 40-pound steam pressure with caustic soda equal to 10 per cent of the weight of the bone-dry stalks and diluted to a 1 per cent solution. The cooked pulp is then washed free of the caustic liquors. These cooking and washing treatments leave a pulp that for all practical purposes may be considered pure cellulose. The pulp is then diluted to a consistency of about 4 per cent and is gelatinized by treatment in a beating engine and a Jordan refiner, two standard pieces of paper-mill equipment, until a jelly-like mass is obtained. This jell is boiled to remove air bubbles, placed in a mold with a perforated bottom and having a plunger top which exerts a pressure of about 0.5 lbs./in.^{2} on the jell, and dried in the mold at about 70° C. The time for drying sheets one-half inch thick is about 10 days. On drying, the maizolith shrinks to about one-fourth of the volume it occupied when molded.

The source of cellulose in the vulcanized fiber process is unloaded, unsized, rag paper. The surface of this paper is gelatinized by running it through a bath of warm zinc chloride, after which the gelatinized paper is rolled up and pressed on a heated hollow drum.[2] The material so formed may be left in the form of a tube or may be cut and pressed into sheets. After removing the fiber from the drum it is washed in progressively weaker solutions of zinc chloride until all traces of chlorides are removed. This washing treatment is very slow, requiring from three weeks for sheets one-eighth inch thick to six months for sheets 2 inches thick. If all traces of chlorides are not removed, a very inferior grade of vulcanized fiber is obtained. After washing, the fiber is dried, during which operation the sheet shrinks to about one-half its size in the wet state.

III. PROPERTIES AND POSSIBLE USES

Some physical properties of maizolith are tabulated in Table 1. The strength tests were made in the civil engineering department of

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[1] U. S. patent 622325.
[2] Allen Rogers, Manual of Industrial Chemistry, 3d ed., p. 1059.

Iowa State College. The specimens for the various tests were of the following dimensions: Compressive strength, a cylinder 0.7 inch long by 0.35 inch in diameter; shearing strength, a bar 0.089 by 1 by 2.33 inches; tensile strength, a rod 3 inches long, turned to 0.375 inch in diameter for 0.5 inch at each end and 0.187 inch in diameter over the remainder of its length; modulus of rupture, a bar 0.375 by 1 by 2 inches.

TABLE 1.—*Some physical properties of maizolith*

	Strength	Electrical properties	Miscellaneous
	Lbs./in.²		
Modulus of rupture.	16,000	Dielectric constant at frequency of 6 kc per second, 7 to 9.	Brinnell hardness, 15 to 30.[1]
Compressive	17,000	Surface resistivity, 7×10^9 ohms per cm² at relative humidity of 45 per cent, temperature of 26° C.	Specific gravity, 1.3 to 1.5.
Shearing	8,000	Volume resistivity, 3×10^9 ohm-cm [3]	Water absorption, 20 to 35 per cent of the original weight in 24 hours.
Tensile	7,000	Phase difference, about 3° at frequency of 6 kc per second.	Oil absorption, 0.09 to 0.13 per cent of the original weight in 24 hours.

[1] This hardness falls in the series for brasses and bronzes.

The electrical tests were made by A. A. Aardal[3] in the physics department at Iowa State College. The dielectric constant and phase difference were measured at a radio-frequency of 6 kc per second, a temperature of 26° C., and a relative humidity of about 45 per cent. The phase difference, which is 3°, indicates that the material will not heat excessively when used as an electrical insulator. The product of the dielectric constant and the phase difference, 24, indicates that the material would be suitable for radio work.

The Brinnell hardness test was made by using a standard Brinnell hardness-testing machine on a test specimen measuring 1 by 1 by 0.25 inch.

The water and oil absorption tests were carried out by immersing pieces of maizolith in water or oil at room temperature. The pieces immersed in water swelled and softened perceptibly after about 15 hours. On drying again they returned to their original hardness, but were apt to develop surface cracks and checks. The samples immersed in oil showed no visible change in physical properties.

Maizolith has been used, on a small scale, in the manufacture of the following articles: Noiseless gears, rods, tubes, refillable fuses, rings, disks, wheels, bearings, screws, and nuts

IV. OPTIMUM CONDITIONS FOR PRODUCING MAIZOLITH

A study of the above-outlined method for the manufacture of maizolith indicates that the factors which probably have the most important bearing on the quality of the maizolith produced are: (1) The raw material; that is, whether the raw material is corncobs or cornstalks; (2) the ratio of caustic soda to the bone-dry weight of stalks; (3) the duration of cooking; (4) the consistency of the pulp at the time of beating; (5) the H-ion concentration at the time of beating; (6) the duration of beating; and (7) the drying temperature. The effect of each of these factors was studied by holding six factors

[3] Unpublished paper by A. A. Aardal, physics department, Iowa State College.

constant and varying the seventh through a definite range. The values for the constant factors were 100 per cent cornstalk pulp; 10 per cent caustic soda based on the weight of bone-dry fiber; enough water to dilute the caustic soda to a 1 per cent solution; a cooking period of three hours at atmospheric pressure; washing until the pH of the pulp was 5.5; beating for six hours in a pebble mill; consistency

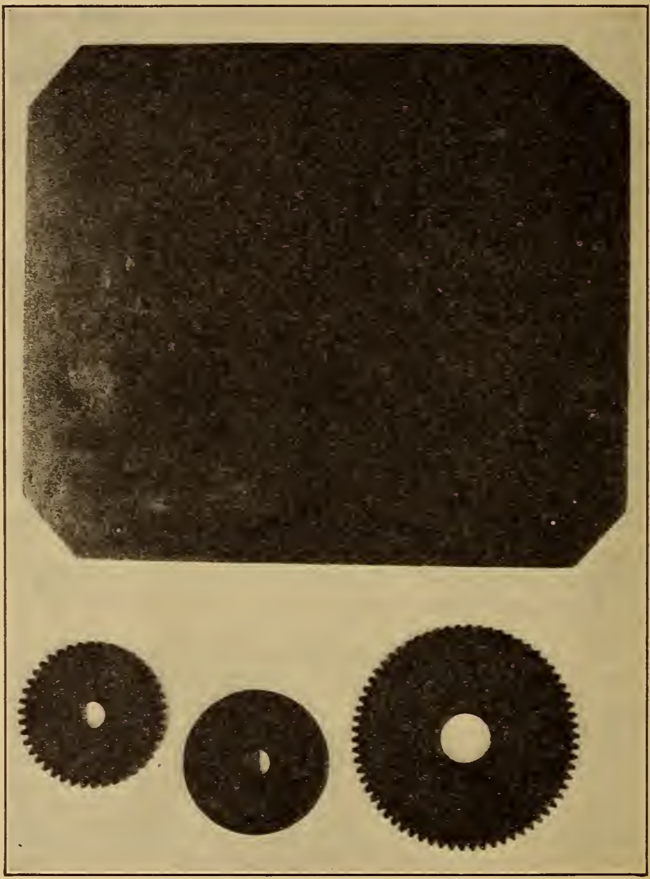

Figure 1.—*Articles made from maizolith*

of 2 per cent in pebble mill; drying temperature of 60° C. The variable factor was then plotted as abscissas against the moduli of rupture of the resulting maizolith samples as ordinates. The effects of the individual factors are shown graphically in Figures 2 to 8, inclusive.

Figure 2 indicates that corncobs and cornstalks give about equally good maizolith but that mixtures of the two yield an inferior product. From this it would seem that the particles of cornstalk fiber are of such a nature that they adhere to each other more firmly than they

do to corncob particles, and that the corncob particles adhere to each other more closely than they do to cornstalk particles.

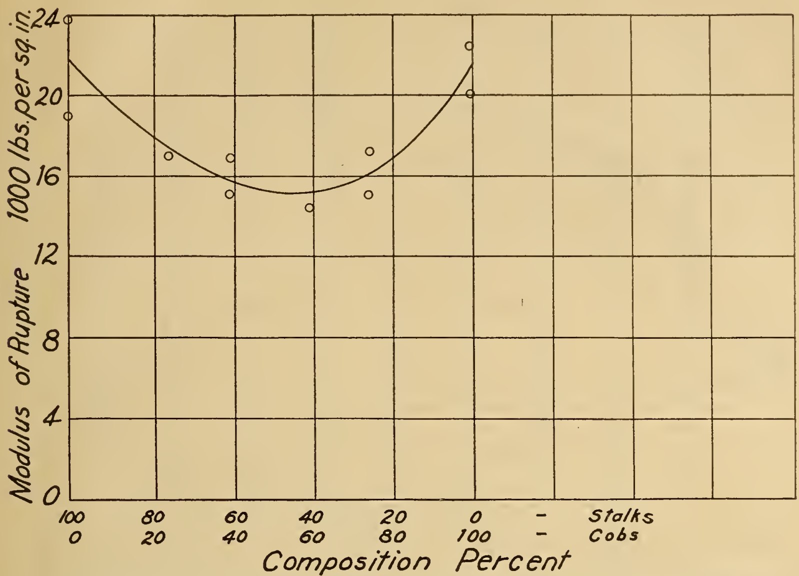

Figure 2.—*Strength of maizolith made from cornstalks and corncobs*

Figure 3 shows that the optimum amount of caustic soda to use in cooking the pulp is from 9 to 11 per cent of the bone-dry weight of

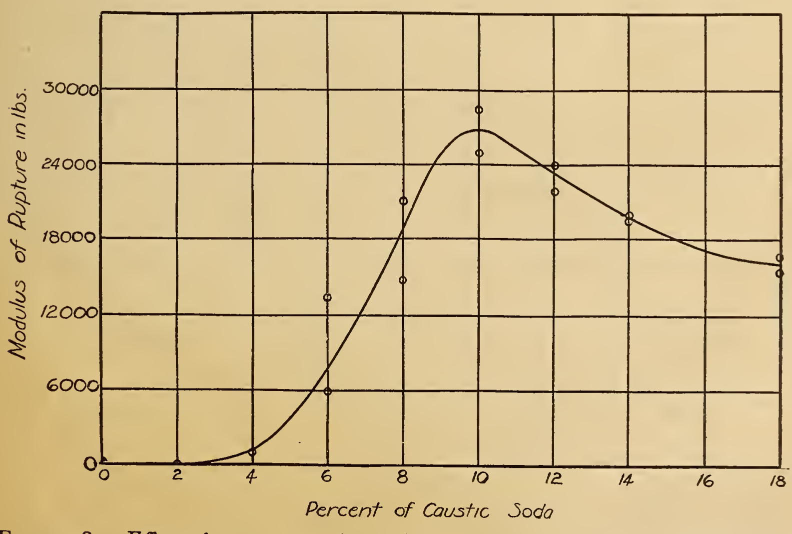

Figure 3.—*Effect of percentage of caustic soda used in cooking on the strength of maizolith*

cornstalks or corncobs. The alkali is diluted to a 1 per cent solution. The time of cooking, according to Figure 4, should be about 16 hours

at atmospheric pressure. Although no corresponding series of runs has been made for pressure cooking, experience in the laboratory at

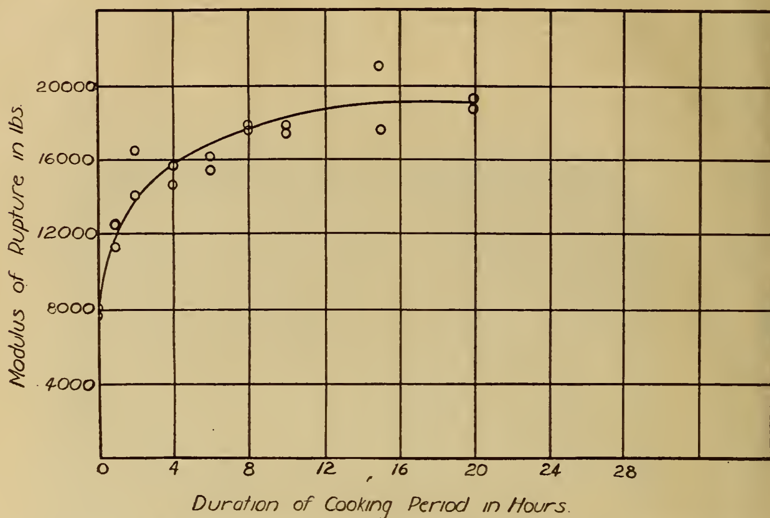

FIGURE 4.—*Effect of duration of cooking on the strength of maizolith*

Ames indicates that three hours' cooking under 40-pound steam pressure is sufficient. The calculations for the amount of water to be

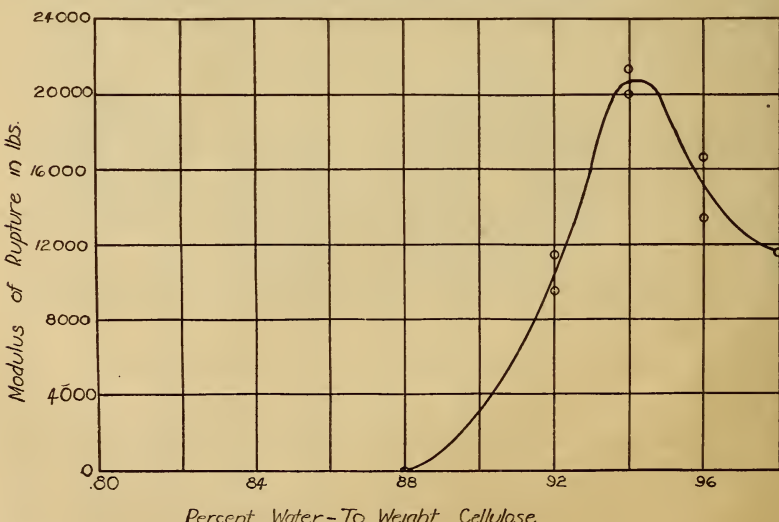

FIGURE 5.—*Effect of consistency of cellulosic materials at time of beating on the strength of maizolith*

added to the pulp, after cooking and washing, to make the pulp up to any given consistency shown in Figure 5, were based on the general

experience that 40 per cent of the total bone-dry weight of the cornstalk or corncob is removed by the cooking and washing operations.

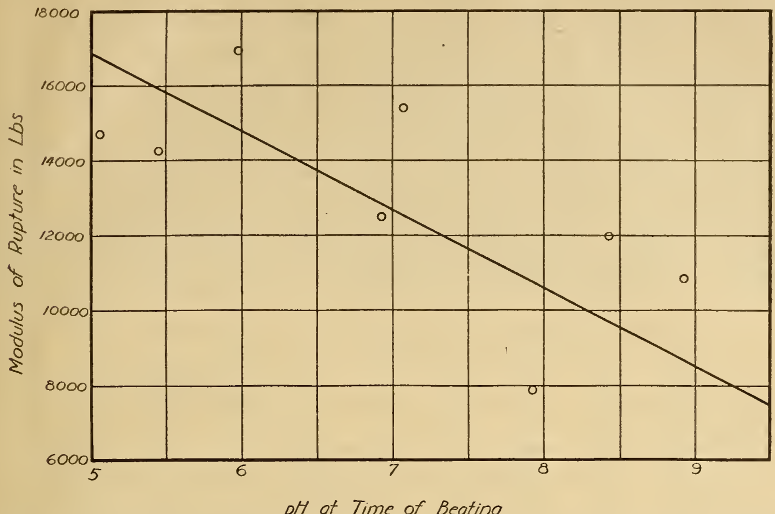

FIGURE 6.—*Effect of the pH of the mixture during the beating process on the strength of maizolith*

Figure 6 indicates that the strength of maizolith increases as the pH at time of beating is decreased from 9 to 5. The optimum pH is evi-

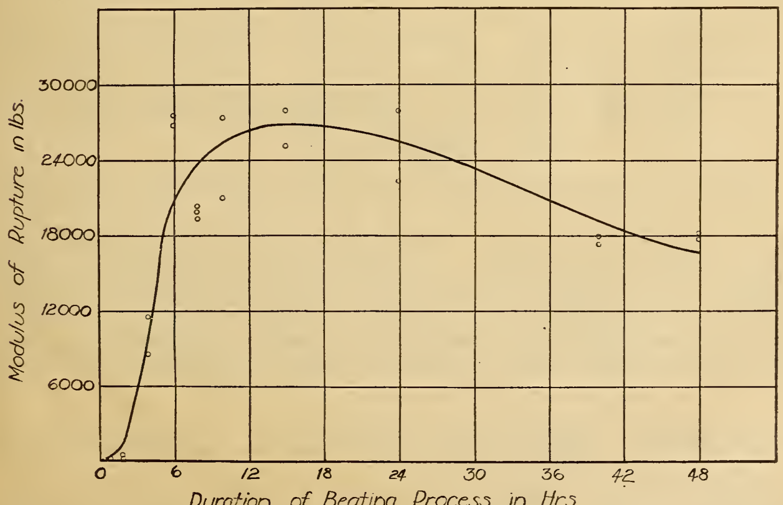

FIGURE 7.—*Effect of duration of the beating process on the strength of maizolith*

dently some value lower than 5; and experiments are now under way to determine this point. The optimum time of beating is 12 to 18 hours as shown by Figure 7. The beating experiments were carried

out in a 4-liter porcelain pebble mill, running at 60 r. p. m. Subsequent experiments have developed a beating method which produces a satisfactory jell in two hours. The cooked and washed pulp is placed in a beating engine, which is connected with a Jordan refiner so that pulp may travel continuously under the beater roll, through the Jordan, and under the beater roll again. The valve connecting the beater to the Jordan is left closed for the first half hour, during which time the pulp is subjected simply to the action of the beater. The valve is then opened and the pulp is given the combined beater-Jordan treatment for one and one-half hours, at the end of which time it is found to be gelatinized to the proper degree for making maizolith. Work is under way at the present time to obtain data which will make it possible to construct a curve for the beater-Jordan machine similar to Figure 7, for the pebble mill. At the present time it is

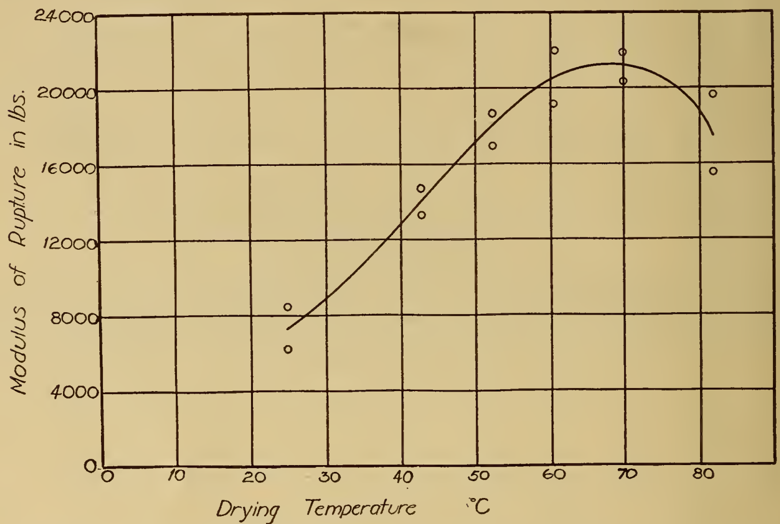

FIGURE 8.—*Effect of the temperature of drying on the strength of maizolith*

known merely that two hours in the beater-Jordan combination gives a pulp which is satisfactory. Whether or not this length of beating treatment is the optimum remains to be worked out. Figure 8 indicates that the best drying temperature is about 70° C. The drying oven was operated at a relative humidity of 75 to 80 per cent. No provision, other than natural convection, was made for circulating air.

V. COST OF PRODUCING MAIZOLITH COMMERCIALLY

Figure 9 shows a suggested layout for a plant to produce 5 tons of maizolith per day. Figuring on the basis of a 50 per cent yield, which experiments have shown may be expected, the plant would require a storage space for 108,000 bales of stalks. This figure assumes a harvesting season of three months; that is, storage space is needed for a 9-month supply of stalks. From the storage piles the baled stalks should be carried by a slat conveyor to a shredder.

Figuring on a 24-hour day this shredder should have a capacity of one-half ton of dry stalks per hour. The shredded stalks should be conveyed to a storage hopper mounted above the digester and arranged so that the stalks can be discharged into the digester. The hopper should have a capacity for a 4-hour output of 2 tons of shredded stalks. The digester should have a capacity of 1,200 cubic feet. The 2 tons of stalks in the digester are cooked under 40-pound steam pressure for four hours with 10 per cent of their bone-dry weight of caustic soda diluted to a 1 per cent solution. From the digester the pulp is pumped to a storage tank having a capacity of 5,000 gallons, where it is diluted to a consistency of 5 per cent. From this tank it is pumped to a continuous suction filter to wash out the material in the pulp which has been dissolved by the treatment in the digester.

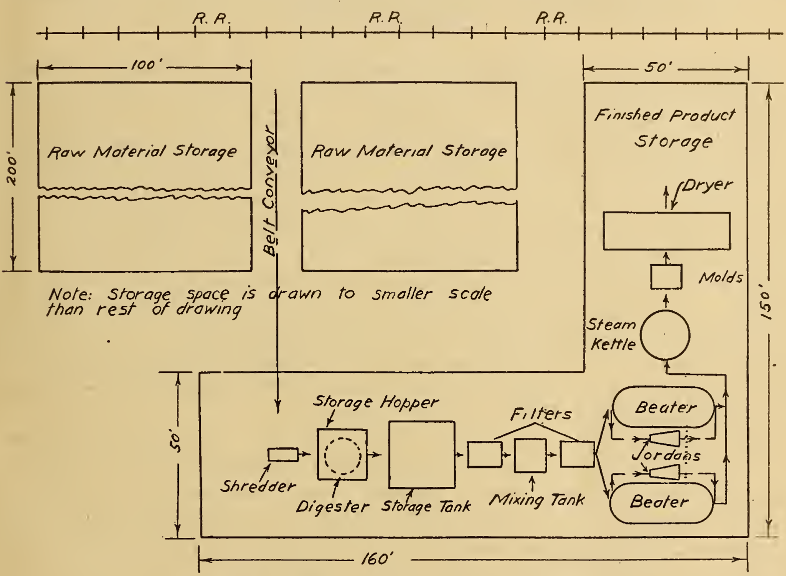

FIGURE 9.—*Flow sheet for maizolith factory*

The pulp from the first filter is broken up, diluted, and pumped to another filter, where the washing operation is completed. After this operation the pulp is given the combined beater and Jordan treatment for two hours as previously described. Two 1,000-pound beaters and two No. 1 Jordans are required to handle the pulp.

From the beaters the pulp passes to a 300-gallon steam kettle in which it is boiled one hour to remove air bubbles. The boiled pulp is poured into a tray 3 by 3 feet by 3 inches deep and dried in an atmospheric shelf dryer. Figuring on a drying time of 10 days, this dryer would have to accommodate 3,560 trays. This could be done in a drier 45 by 32 feet by 12½ feet high, if the drier shelves were 44 feet long by 31 feet wide, and the shelves were spaced 6 inches apart.

Based on the process outlined above for making maizolith, quotations have been secured from manufacturers of the required equipment which indicate that a plant to produce 5 tons of the material per day would involve a capital layout of about $325,000. On the

basis of this capital requirement and estimated cost for the various steps in the process, maizolith could be produced for about $140 per ton. This figure includes charges for raw material, cost of production, depreciation of equipment, and manufacturing overhead.

The figure of $140 represents the cost per ton of maizolith as it comes from the drier in a rather rough form. If a charge of 75 per cent, or $100, is added for the cost of machining, a total of $240 is obtained as a very rough estimate of the cost of a ton of the finished product.

Maizolith, manufactured as a by-product of the cornstalk insulation board industry, or the corncob adhesive industry, could probable be produced at a figure lower than $240 per ton.

VI. MAIZOLITH AS A BY-PRODUCT FROM OTHER INDUSTRIES

In the process of making an insulating wall board from cornstalks, it may be necessary before forming the board to remove the fibers which are smaller than a certain size. If this is the case these small fibers would make an excellent raw material for the manufacture of maizolith. Experiments conducted at the experimental insulating board plant at Ames indicate that a plant producing 150,000 square feet of insulating board per day would furnish enough fine material to make 3,000 pounds of maizolith per day. This fine material would be put through the same process as that outlined above for the cornstalks except that instead of being passed through the shredder they would be run through a filter and then to the digester. This modification of the process would then reduce the cost of the raw material and the shredding and add the expense of filtering and hence make the total cost less than that outlined above.

The process of extracting an adhesive material from corncobs is one which gives promise of soon becoming commercial.[4] [5] In this process corncobs are steam digested for a short time. The liquor which is drained off shows good adhesive properties when concentrated. This treatment leaves 55 per cent of the cob as a soft residue, in ideal condition for conversion into maizolith. This process would be the cheapest of the three, since the cost of raw material and beating would be reduced, and expense of the shredding eliminated.

Since the demand for a low-priced material which combines strength, hardness, and electrical insulating properties is continually increasing, it seems probable that maizolith, which can be produced cheaply, and which has the desired properties, may soon play an important part in supplying this need.

WASHINGTON, December 18, 1929.

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[4] T. R. Naffziger, Iowa State College, The Manufacture of an Adhesive Material from Corncobs, unpublished thesis; 1928.

[5] Frederick B. LaForge, The Simultaneous Production of Pentosan Adhesives and Furfural from Corncobs and Oat Hulls, J. Ind. Eng. Chem., **16**, p. 130; 1924.

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